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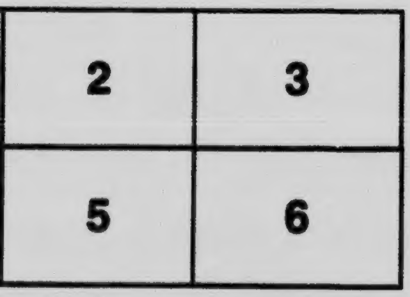

UNIVERSITY OF TORONTO STUDIES

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No. 78: LOSS OF CARBON DURING SOLUTION OF STEEL IN POTASSIUM CUPRIC CHLORIDE, BY E. P. MOORE AND J. W. BAIN

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LOSS OF CARBON DURING SOLUTION OF STEEL IN POTASSIUM CUPRIC CHLORIDE.

BY

E. P. MOORE AND J. W. BAIN.

LONDON:
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Meeting held at Toronto on Thursday, May 16, 1908.

PROF. W. H. ELLIS IN THE CHAIR.

LOSS OF CARBON DURING SOLUTION OF STEEL IN POTASSIUM CUPRIC CHLORIDE.

BY E. P. MOORE AND J. W. BAIN.

There has been much uncertainty as to whether carbon is lost by the evolution of gaseous hydrocarbons during the solution of steel in acid potassium cupric chloride solution when [illegible] method is used for the determination of carbon in [illegible] metal. Dillner(1) remarks, "As is well known and as has been pointed out on several occasions by various authorities, there is always an evolution of hydrocarbon gases when steel dissolves in copper chloride, which escape"; accounting in this way for the lower results obtained from the indirect processes. He also refers to some observations made by Ledebur(2), but unfortunately the publication was inaccessible. Arnold(3) states that he has seen distinctly bubbles rising through the liquid during the solution of steel in copper chloride.

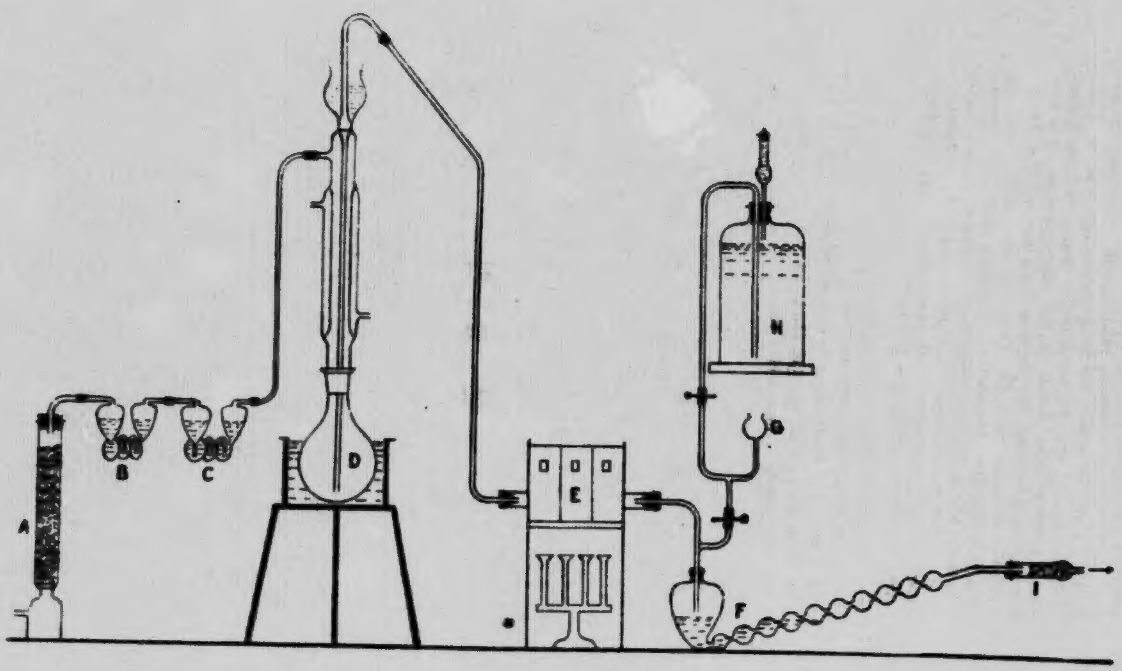
The problem has hitherto been attacked from the following standpoint. Samples of steel are dissolved in various solvents of various concentrations and the carbon estimated as usual. The highest percentage obtained is selected as a standard and the discrepancies between this and the other results are ascribed to losses during solution. True, these results are confirmed by determinations made by the volatilisation of the iron by chlorine or by direct combustion, but even in these cases the uncertainty of a numerical difference between two results, each burdened with experimental errors, is obvious. For these reasons it appeared to be desirable to determine, by a direct process if possible, whether any carbon was lost during the solution of iron or steel in the solvent so commonly used—potassium cupric chloride.

The following apparatus was used (see figure):—A, A drying tower filled with potassium hydroxide in sticks. B, An absorption bulb filled with potassium hydroxide solution. C, An absorption bulb filled with barium hydroxide solution. D, A Rheindorf(4) flask for estimation of carbon, immersed in a water bath maintained at a temperature of 65° C. E, A piece of combustion tubing filled with cupric oxide, heated to redness in a small gas furnace. F, A ten bulb absorption apparatus charged with barium hydroxide. G, A small thistle tube sealed to a glass T as shown. H, A stock bottle containing barium hydroxide. I, A guard tube filled with soda-lime. The introduction of barium hydroxide into the absorption bulb is the only feature which calls for remark. A current of air free from carbon dioxide having been drawn through the bulb, F, for some time, a suitable quantity of barium hydroxide solution was siphoned over, being clarified in transit by passing through a filter of ignited asbestos (not shown). In the first experiments a slight precipitate formed on the inner surface of the delivery tube which protrudes into the bulb, F; by pouring into the funnel, G, a small quantity of recently-boiled distilled water and allowing it to wash down, it was found that this could be entirely avoided.

The flask, D, was charged with a solution of potassium cupric chloride made by dissolving 300 grms. of the salt in 1 litre of water and adding 75 c.c. of concentrated hydrochloric acid. Blank tests were made by placing in the flask, D, the quantity of potassium cupric chloride solution usually employed, and after aspirating for some time to clear the apparatus of carbon dioxide, filling the bulb, F, with barium hydroxide solution, and proceeding to heat the flask, D, to 65° C. After aspirating for two hours, in no case was there more than a very faint cloud of barium carbonate to be observed in the tube, F; efforts were made to collect and weigh this precipitate, but without success. Three grms. of steel were then placed in the solution flask, 200 c.c. of potassium cupric chloride solution added, and the water bath maintained at a temperature of 65° C.; air was also slowly aspirated through the apparatus, the cupric oxide having been previously brought to red heat. When the steel had dissolved completely, the ten bulb tube was removed, and a stopper carrying a soda-lime tube placed immediately in the larger end, while the other opening was connected by rubber tubing to the glass tube in the cork of a filtering funnel. The barium carbonate was transferred as completely as possible to the filter and washed with recently-boiled distilled water; the absorption tube and the filter were then treated with dilute hydrochloric acid, the asbestos filtered off, and the barium precipitated as sulphate and weighed.

Two samples of steel were employed; the one containing 0·653 per cent. of carbon, the other 1·18 per cent. Four determinations were made on each of these, 3 grms. being used in each case; the results are expressed as grms. of carbon escaping from the flask.

	0·653 per cent. Steel.	1·18 per cent. Steel.
	grm.	grm.
	0·00176	0·00115
	0·00112	0·00121
	0·00142	0·00100
	0·00159	0·00103
Mean	0·00147	0·00110
Loss on 1 grm.	0·0005	0·0004


A
B
C
D
E
F
G
H
I

In the case, therefore, of the 0·653 per cent. steel, a quantity of carbon corresponding to 0·05 per cent. has been lost during solution, making the true percentage 0·703; with the other the true percentage will be 1·22.

These losses are comparatively large, although not much greater than some of the discrepancies between the results obtained by various members of the International Committee. In view of the difficulties in manipulation the authors do not profess that their figures represent the actual loss, but they believe that they have proved that a loss does take place under the conditions given above, and also that the amount approximates fairly closely to the figures given.

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(1) J. Iron and Steel Inst., 66, 255.

(2) Verein zur Beförderung der Gewerbfleisses, Vols. 6, 7.

(3) Steel Works Analysis, 2nd ed., p. 29.

(4) Zeits. angew. Chem., 1904, 467.

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